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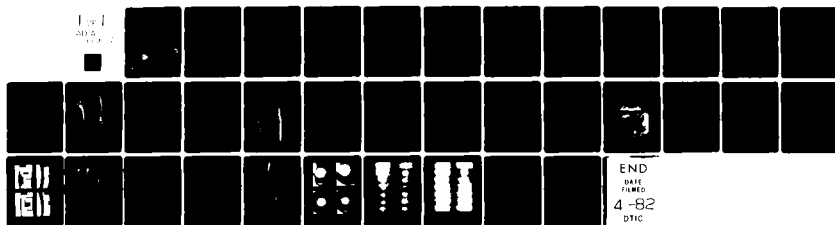
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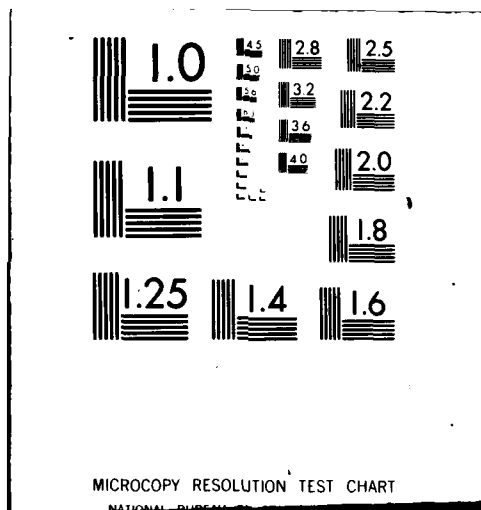
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Final Report

PROGRAM TO COMPARE
DEUTERATED LUBRICANT WITH
SYNTHETIC HYDROCARBON LUBRICANT

Subcontract No. 80-7001

18 September 1981

by

Erik Gelotte
and
Edward Kingsbury



The Charles Stark Draper Laboratory, Inc.
Cambridge, Massachusetts 02139

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<p>A program was conducted under subcontract to Geo-Centers Inc., Newton Upper Falls, MA (subcontract No. 80-7001) to evaluate, by direct comparison, the performance of a synthetic hydrocarbon lubricant with a modified version in which the hydrogen had been replaced by deuterium. The program was conducted in two tasks as follows:</p> <p>Task I: Operating Bearing Evaluation, and</p> <p>Task II: Controlled Slip Evaluation.</p>		

It was found in Task I that at each test temperature the deuterated grease performed better, that is, ran for a longer time before the bearing torque doubled, than the hydrogenated grease.

In the controlled slip evaluation a qualitative difference in degradation rates between the hydrogenated and deuterated oils was observed for the flooded, high slip, low pressure ball-to-ball contacts in these tests. The deuterated oil showed lower degradation rates. No difference could be shown for the high pressure, low slip ball-to-race contacts.

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Final Report

Program to Compare Deuterated Lubricant with
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Approved:



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SECTION 1

TASK I: OPERATING BEARING EVALUATION

1.1 Task I Objective

The objective of Task I was to obtain a direct comparison between the life of standard off-the-shelf bearings running under controlled conditions when lubricated with a hydrogenated and a 97 percent deuterated version of the same synthetic hydrocarbon.

The comparisons were to be made at each of three temperatures by measuring the running time to failure (defined as that point at which the bearing package torque doubles) of six sets of bearings with each lubricant. (H = hydrogenated, D = 97 percent deuterated lubricant.) Refer to Table 1-1.

Table 1-1. Lubricant test plan.

TEMPERATURE	LUBRICANT			
	GREASE		OIL	
	H	D	H	D
400°F	6 sets	6		
350°F	6	6	6	6
250°F	6	6		

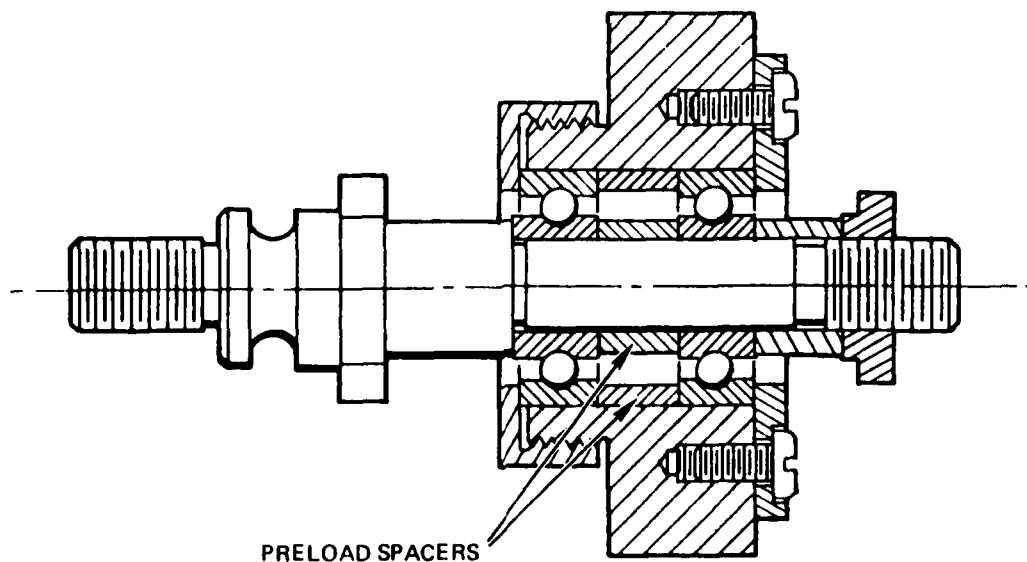
The bearings were to be run in air at 12,000 r/min at a preload of 5 to 7 lbs (Hertz-stress range of 108,000 to 152,000 lb/in²) and no externally applied load.

1.2 Summary

At each test temperature the deuterated grease performed better, that is, ran for a longer time before the bearing torque doubled, than the hydrogenated grease.

1.3 Test Bearings and Preparation

Standard R-4 size bearings, purchased from KuBar Bearings, Inc., Cambridge, Massachusetts [SRB4HHK35LDZD(7)], were received dry from the manufacturer and without the shields assembled. Before running in bearing cartridges (Figure 1-1) in which the preload could be set by suitable manipulation of the lengths of the preload spacers, bearings were processed as follows:



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Figure 1-1. Bearing cartridge.

- (1) Ultrasonic cleaned for 5 minutes in successive baths of toluene, Freon, and methanol; vacuum dried between each, followed by a dip in a 50/50 mixture of methanol and acetone.
- (2) Weighed (with shields and snap-rings).
- (3) Lubricated by immersion in a 50/1 mixture of hexane and SRG-60.
- (4) Calibrated (torque measured at 1 r/min on low-speed dynamometer at 5, 7, and 9-lb load.
- (5) Offsets measured.
- (6) Assembled as pairs with appropriately lapped spacers into cartridges (refer to Figure 1-1), and preload verified on low speed dynamometer. Spacers relapped if necessary.
- (7) Bearing cartridges disassembled and bearings recleaned (Step 1).
- (8) 35 ± 5 mgm of grease applied to each side of each bearing before shields and snap-rings installed. Each bearing rotated under no load to distribute lubricant.
- (9) Reassembled into bearing cartridges.

1.4 Testing

Fixtures were built that could mount six bearing cartridges and maintain them at the test temperature while the shafts were rotated at 12,000 r/min with dc permanent-magnet motors whose armature current was measured to determine running torque (of motor plus test bearings). The torque constants of the motors were $2.27 \frac{\text{oz-in}}{\text{amp}}$ ($\pm 3\%$).

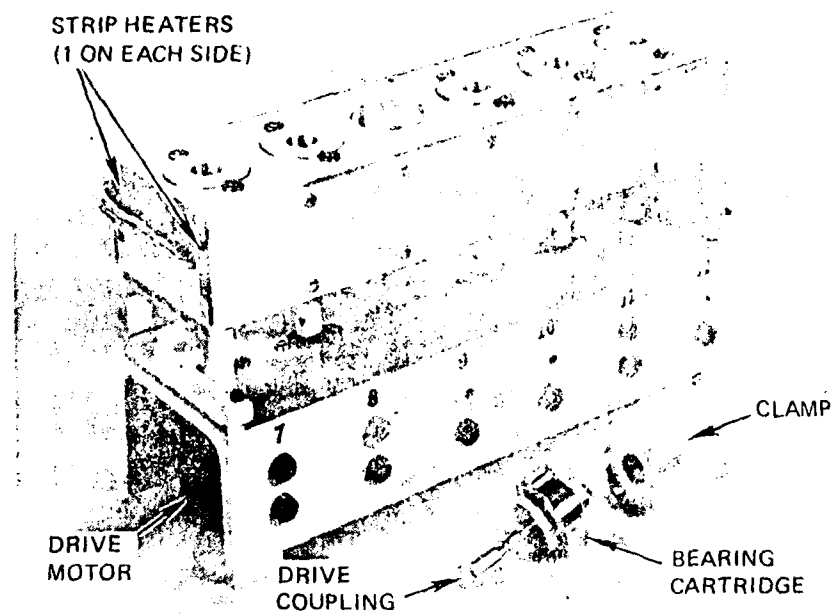
Prior to installing the bearing cartridges in the running fixture (Figure 1-2), the motors were run for a short time with the fixture at test temperature in order to determine the running torque of the motors alone. The bearing cartridges were then installed and the test was begun.

Originally the intent was to measure and record the motor current every two hours during the first few running hours to obtain the "settled" value of the bearing torque and to take readings twice a day thereafter. The first set to be run was the 400°F test on the hydrogenated grease. All six packages failed during the night between the first and second day and indicated the need for supplementing the data-taking with continuous strip-chart recordings. This was done for all subsequent tests.

A "failure" current level was obtained for each test-set by summing the "motor-alone" current and twice the settled test-bearing current.

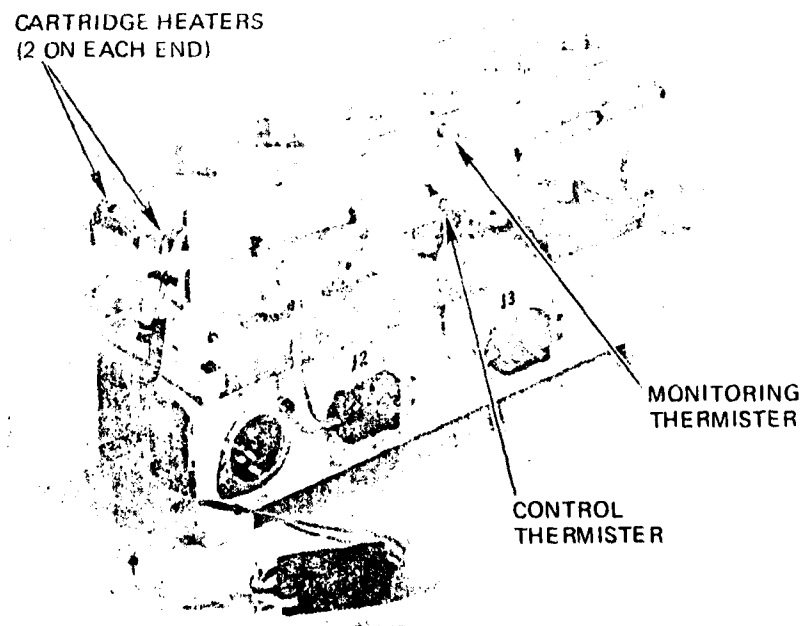
In addition to the motor-current strip-charts, sample sections of which are shown in Figures 1-3 through 1-8, currents and temperature were measured and recorded each working day during the test, and minor speed (motor voltage) and temperature adjustments were made as required.

Since changes in the drive-motor bearings were reflected in the current data, it was necessary to verify that an indicated failure was not a motor failure. This was done in each case by uncoupling the test bearings and remeasuring the current drawn by the motor alone. If the motor had failed, the test bearings were driven by a replacement motor. Despite the performance specified for the commercial motors, most of them failed in less time than the specifications indicated and additional motors had to be obtained to complete the testing program.



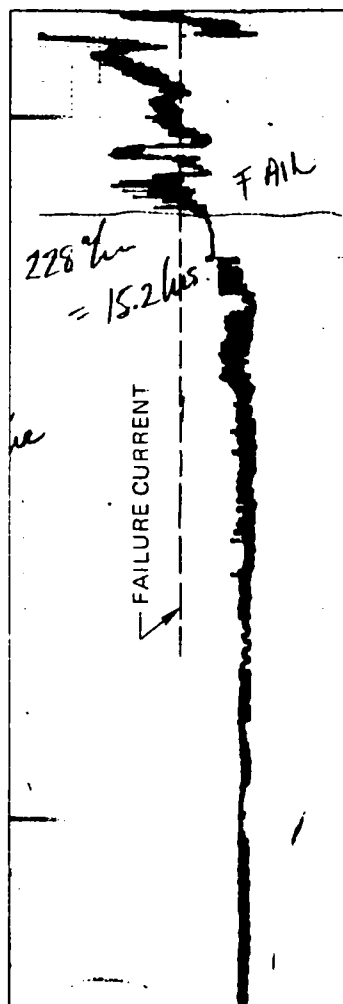
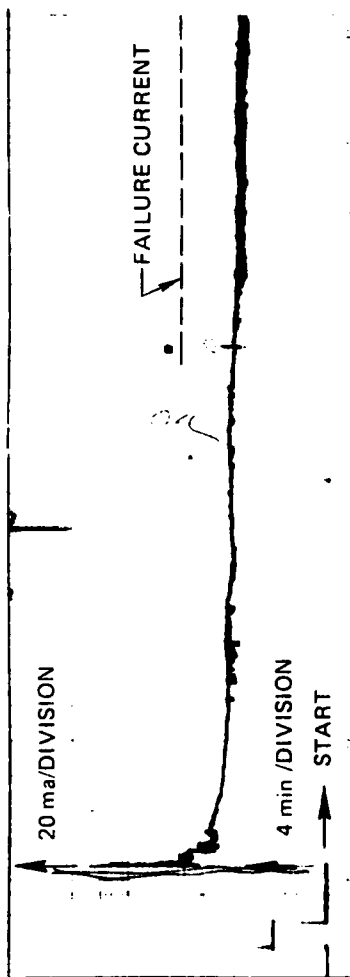
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Figure 1-2(a). bearing running fixture (front).



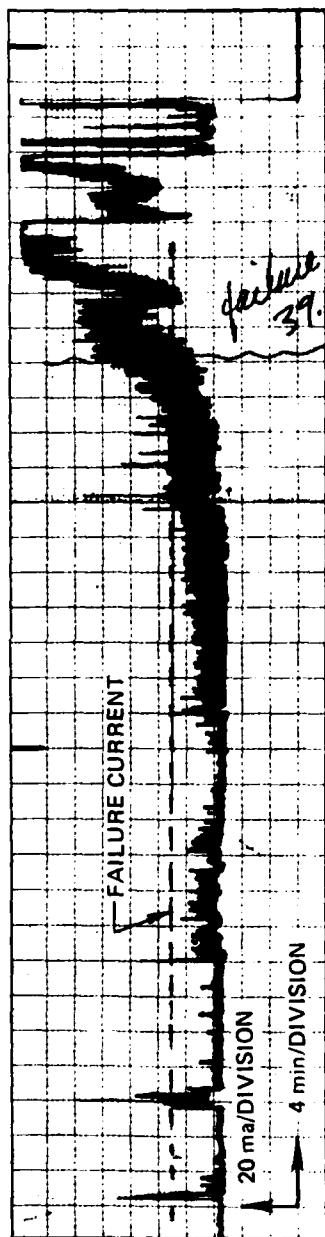
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Figure 1-2(b). bearing running fixture (rear).



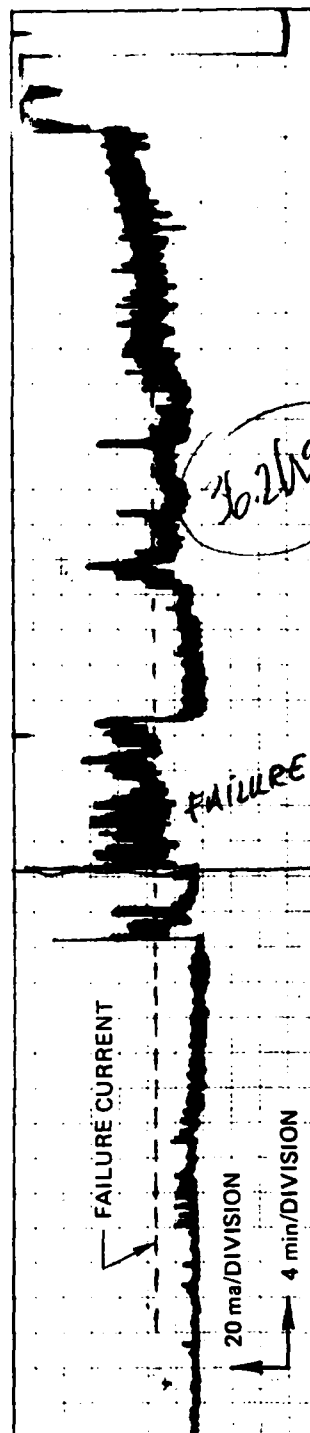
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Figure 1-3. Hydrogenated grease/400°F set no. 6.



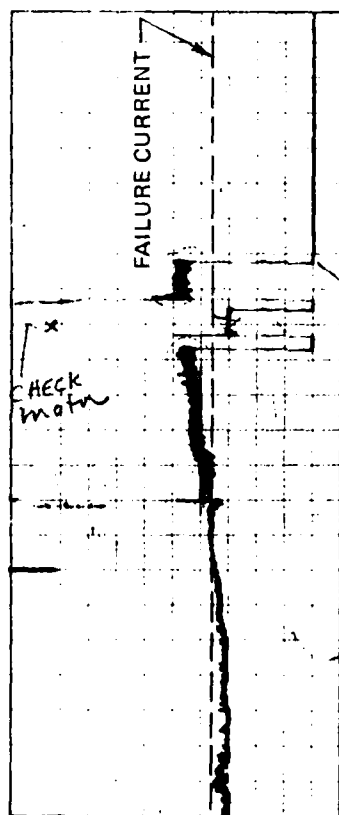
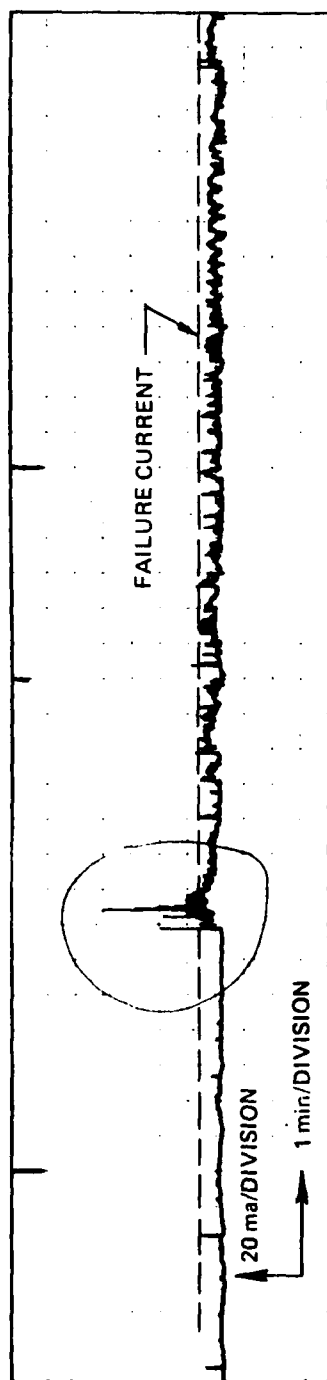
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Figure 1-4. Hydrogenated grease/350°F set no. 6.



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Figure 1-5. Hydrogenated grease/350°F set no. 5.



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Figure 1-6. Deuterated grease/400 F set no.2.

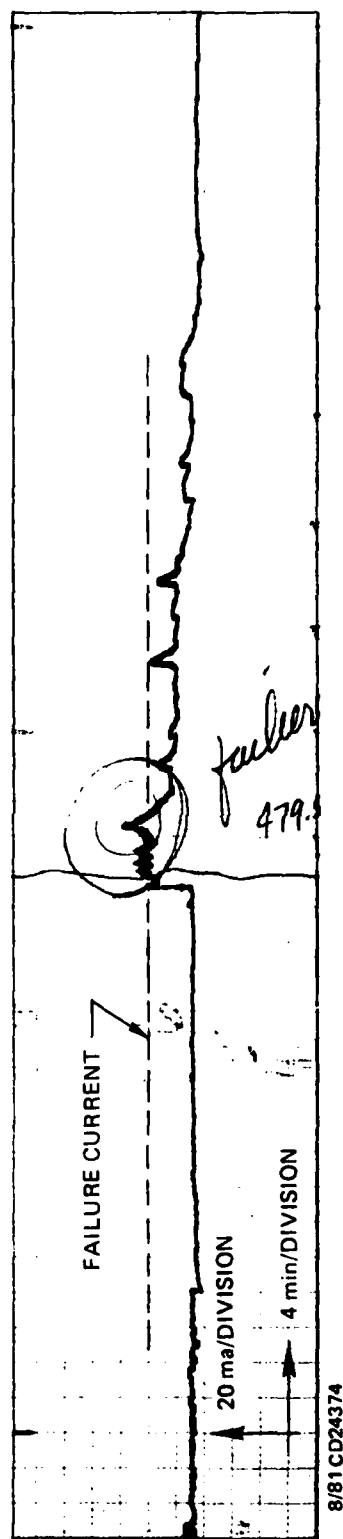
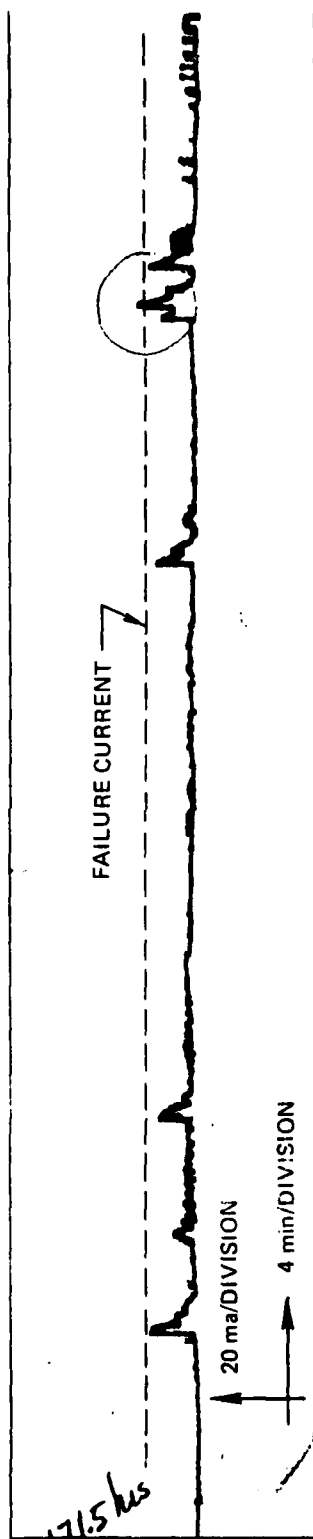
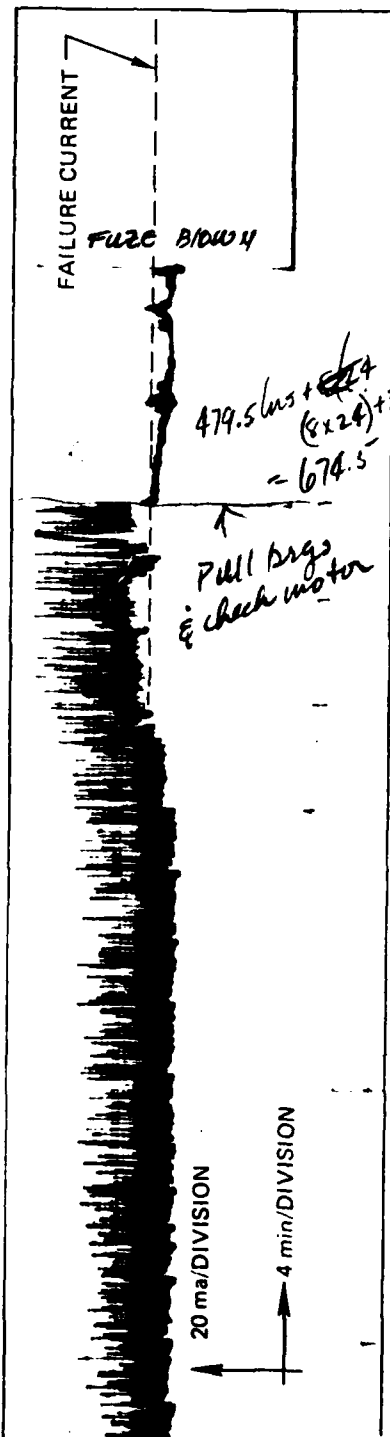
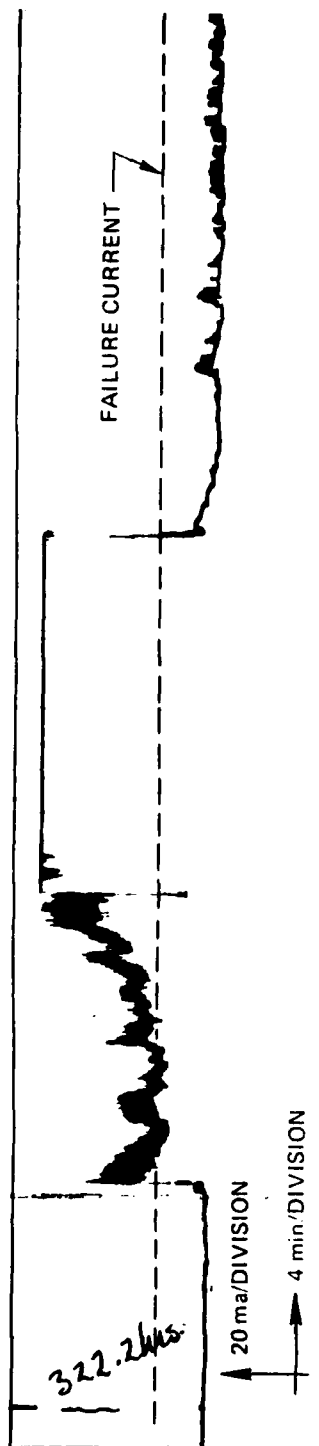


Figure 1-7. Deuterated grease/250°F set no.4.



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Figure 1-8. Deuterated grease/250°F set no.4.

Figure 1-3 illustrates a typical current (torque) history of a test-set. The current is initially high, then settles as the grease is distributed. The failure is well-defined. Similarly, in Figure 1-4 the failure is fairly well-defined although some short-term spikes occur before the average torque rises unquestionably above the failure criterion level. Many of the bearing packages showed very erratic torque behavior (see Figures 1-5 through 1-8). An extreme example is shown in Figures 1-7 and 1-8, where a test-set ran erratically for 674 hours. In view of such results, an expansion of the failure criterion was deemed necessary. It was decided to consider failure as having occurred at that point where the bearing torque doubled without decreasing for 20 minutes (minimum).

The tests were run starting with the high temperatures (e.g., 400°F). The hydrogenated lubricant was run first at each temperature. Unfortunately, the temperature controller stuck closed shortly after the start of the hydrogenated/250°F set. This raised the temperature to at least 562°F and invalidated these test results. The test was restarted with fresh bearings after adding an over-temperature cut-out to the control circuit.

Because of the loss of these bearings and the desire to rerun the first test (hydrogenated grease/400°F) with strip-chart recordings of the motor currents, it was not possible to run the test on the oil samples without obtaining more bearings. In addition, other modifications to the bearings, such as dam-grinding and replacement of the metal ribbon retainers with porous retainers, were deemed necessary in order to properly test the oils.

Five of the six bearing sets with deuterated grease at 250°F had failed when it became necessary to stop the sixth set in order to repeat the hydrogenated/400°F test. This set had not shown any signs of failure and was still running at 1854 hours. The longest time to failure among the five failed sets was 1356 hours.

1.5 Results and Discussion

A compilation of the test results is shown in Table 1-2 and the average times to failure with standard deviations are shown in Table 1-3. There can be little doubt that the deuterated grease produced an increase in time-to-failure and that the improvement appears greater at the lower test temperatures.

Many of the test sets did not show a smooth monotonic torque increase (hash and/or average level) to failure. This introduces some uncertainty into interpretation of the results but does not change the basic conclusion.

In addition to tabulating the failure times, each failure in Table 1-2 is characterized as being one of the following types:

- (a) essentially monotonic torque increase to failure
- (b) other - i.e., erratic

The spread in the data suggests that the results reflect non-lubricant-related factors such as a significant variation in the quality of the bearing parts themselves. In addition, the relatively short failure times of most of the bearings suggest a problem with the bearings themselves.

Although the scope of the program did not include any diagnostic disassembly of the bearings, it was noted that each failed bearing pair had the characteristic appearance of lubricant deterioration with large amounts of "dry" debris visible coming out from under the shields. The sixth set in the deuterated grease/250°F series, which had not failed when it was stopped, looks good and should be carefully disassembled and inspected.

Table 1-2. Test results.

LUBRICANT/TEMPERATURE	DATE STARTED	FAILURE TIMES (Hours)
Hydrogenated grease/400°F	11/19/80	All 6 sets failed in 2-12 hours (no recording)
Hydrogenated grease/400°F (2nd set)	6/23/81	12.7(a), 4.9(a), 18.1(a), 22.2(b), 13.2(a), 15.2(a)
Deuterated grease/400°F	12/11/80	<3.0, 30.3(b), 29.0(b), 32.3(a), 31.0(b), <1.0
Hydrogenated grease/350°F	1/16/81	46.2(b), 48.2(b), 42.2(a), 36.2(b), 50.8(a), 39.0(b)
Deuterated grease/350°F	1/29/81	53.3(a), 82.6(a), 144.6(a), 86.0(a), 102.6(a), 28.3(b)
Hydrogenated grease/250°F	2/12/81	8.5(b), 116.1(b), 217.2(b), 437.4(a), 252.3(a), 645.8
Deuterated grease/250°F	3/23/81	492.2(b), 908.5(b), 813.9(a), 210.2(b), 1356(b), 1853.8 (and still running when stopped)

NOTES: (a) essentially monotonic torque increase to failure

(b) other (i.e., erratic)

Table 1-3. Average time to failure.

Temperature (°F)	Average Time to Failure (hrs)		Standard Deviation (hrs)	
	Hydrogenated Grease	Deuterated Grease	Hydrogenated Grease	Deuterated Grease
400	14.4	21.1 (30.7)*	5.8	14.8 (1.4)*
350	43.8	82.9	5.6	40.2
250	279.6	939.2**	229.7	593.0**

* excluding 2 immediate failures

** One set still running without signs of failure when test was terminated at 1853.8 hours

1.6 Conclusions

The bearings lubricated with deuterated grease performed better than those lubricated with the hydrogenated grease. The improvement appears greater at the lower test temperatures.

1.7 Recommendations

Further testing of bearings should take into consideration:

- (1) Refinement of the failure criterion
- (2) Screening of the bearings before testing the lubricant
- (3) Altering the test set-up to uncouple the drive-motor-bearing performance from that of the test bearings. One way to accomplish this would be to lubricate the motor bearings themselves with the test lubricants.

SECTION 2

TASK II: CONTROLLED SLIP EVALUATION

2.1 Task II Objective

Mechanical activation of chemical degradation reactions in rolling bearing lubricant has been suggested.^{(1,2,3)*} The high shear rates and high pressures in elastohydrodynamic (EHD) contacts can supply the energy required to activate oxidation or polymerization reactions. With well controlled rolling conditions a bearing test can be used to distinguish lubricant degradation susceptibility amongst lubricants. Thus differences between typical mineral oil lubricants and two pure substance hydrocarbons (and possibly between similar oils of different viscosity) have been reported.⁽⁴⁾ It has been shown elsewhere⁽⁵⁾ that thermally activated reactions appear slower in deuterated lubricants. It was thought that a bearing test might also show a difference in mechanically activated reactions. Thus the present experiments sought to compare two fluids for breakdown when run under well-defined conditions in a small angular contact ball bearing. The fluids were the same except that 97 percent of the hydrogen in the first was replaced with deuterium in the second.

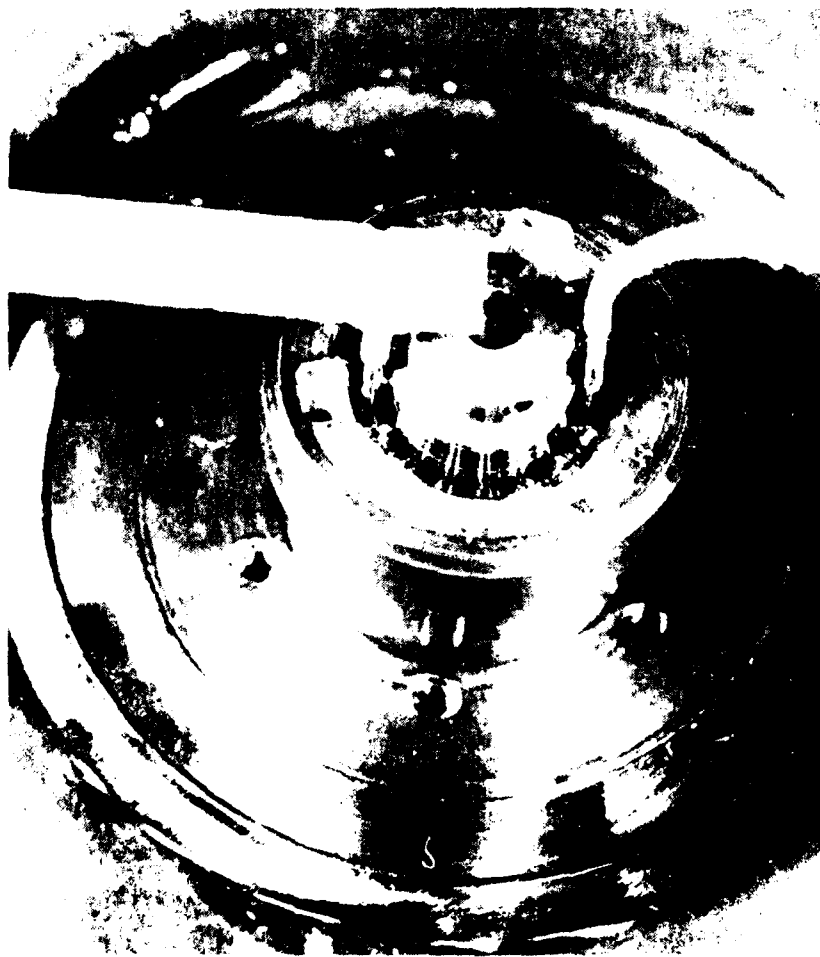
2.2 Summary

A qualitative difference in degradation rates between the hydrogenated and deuterated oils was observed for the flooded, high slip, low pressure ball-to-ball contacts in these tests. The deuterated oil showed lower degradation rates. No difference could be shown for the high pressure, low slip ball-to-race contacts.

* Superscript numerals refer to similarly numbered items in the List of References.

2.3 Test Arrangement

A bearing can be run with its races counter rotating at such rates that the ball centers are stationary. An apparatus suitable for this operational mode is shown in Figure 2-1 and its theoretical background is discussed in reference (6). If the bearing is run without a retainer a controlled initial oil charge can be used as an independent variable to set film thickness (hence shear rate).



TSA 1828

Figure 2-1. Counter rotation fixture.

All tests were run using the same two races (KuBar SR4) in a retainerless full ball complement configuration. A new ball set was used for each test; one ball in each set was given a through hole to identify its spin axis; the races were mechanically and the chemically cleaned between tests according to a standard procedure. Running conditions and nominal contact parameters for all tests are as shown in Table 2-1.

Table 2-1. Test conditions and nominal contact parameters.

	INNER RACE	OUTER RACE	BALL	ASSEMBLY
Element Rate	140 Hz	-95 Hz	-525 Hz	235 Hz
Orbit Rate	NA	NA	0	0
Axial Load	NA	NA	NA	40N
Ave. Hertz Stress	$1.03 \times 10^9 \text{ N/m}^2$	$0.85 \times 10^9 \text{ N/m}^2$	NA	NA
Semi Major Hertz Width	$1.14 \times 10^{-4} \text{ m}$	$1.09 \times 10^{-4} \text{ m}$	NA	NA
Semi Minor Hertz Width	$2.54 \times 10^{-5} \text{ m}$	$3.05 \times 10^{-5} \text{ m}$	NA	NA
Hertz Area	$9.1 \times 10^{-9} \text{ m}^2$	$1.04 \times 10^{-8} \text{ m}^2$	NA	NA

In air at 20°C

Assuming nominal lubricant properties of 0.2 N s/m^2 viscosity (constant with pressure), a molecular weight of 515, and nominal film parameters of 0.01 m/s slip and $2.5 \times 10^{-5} \text{ cm}$ thickness, an energy density of $1.8 \times 10^{12} \text{ J/kmole}$ is estimated inside the Hertz zone, compared with an activation energy of 10^8 J/kmole for the carbon-to-carbon bond. This is an underestimate since high pressure viscosity is enhanced and typical starved film thicknesses are reduced from the assumed values in the test situation.

2.3.1 Starved Tests

Changes in the amount and state of a fluid lubricant within the EHD contacts of a starved bearing can be followed by measuring its basic speed ratio. This characteristic number is defined as ball spin rate over bearing total speed;⁽⁷⁾ both can be measured with precision. Ball spin rate is set by race-lubricant-ball coupling, which improves as fluid is lost from the EHD contacts. An increasing basic speed ratio is thus associated with worsening fluid conditions in the nip.^(2,6,8)

Previous studies have shown that if an R4 bearing is supplied with a small amount of lubricant (e.g., 100×10^{-6} gm) that is not replenished during running, its basic speed ratio shows an initial quick drop through a minimum (Region I), then a gradual slow increase (Region II), and then a rapid rise to failure (Region III). (Refer to Figure 2-2.)

The initial transient is unexplained. The increase in Region II is thought to be caused by loss of oil from the EHD contacts (a) by side flow within the Hertz zone, or (b) by (slow) degradation reactions from fluid to solid. As fluid is lost the EHD film thins, increasing shear rate within the lubricant past a (hypothetical) critical value into Region III, with failure (as evidenced by disturbances in the ball spin axis) quick to follow. If carefully monitored, the failure can be limited to the lubricant (no metal damage). The solid detritus can be removed from the races which can be reused, thus removing questions of metallurgy from these tests. This sequence is shown in Figure 2-3, in photos of the test races before (a), after (b), and after cleanup (c) from the run of Figure 2-2. The lubricant breakdown material in Figure 2-3b was formed almost entirely during Region III of Figure 2-2 (refer to section 2.3.2 on flooded tests). The absence of gross metal damage in this failure is seen by comparing 2-3a and 2-3c, which show the same part of both races, before and after the failure.

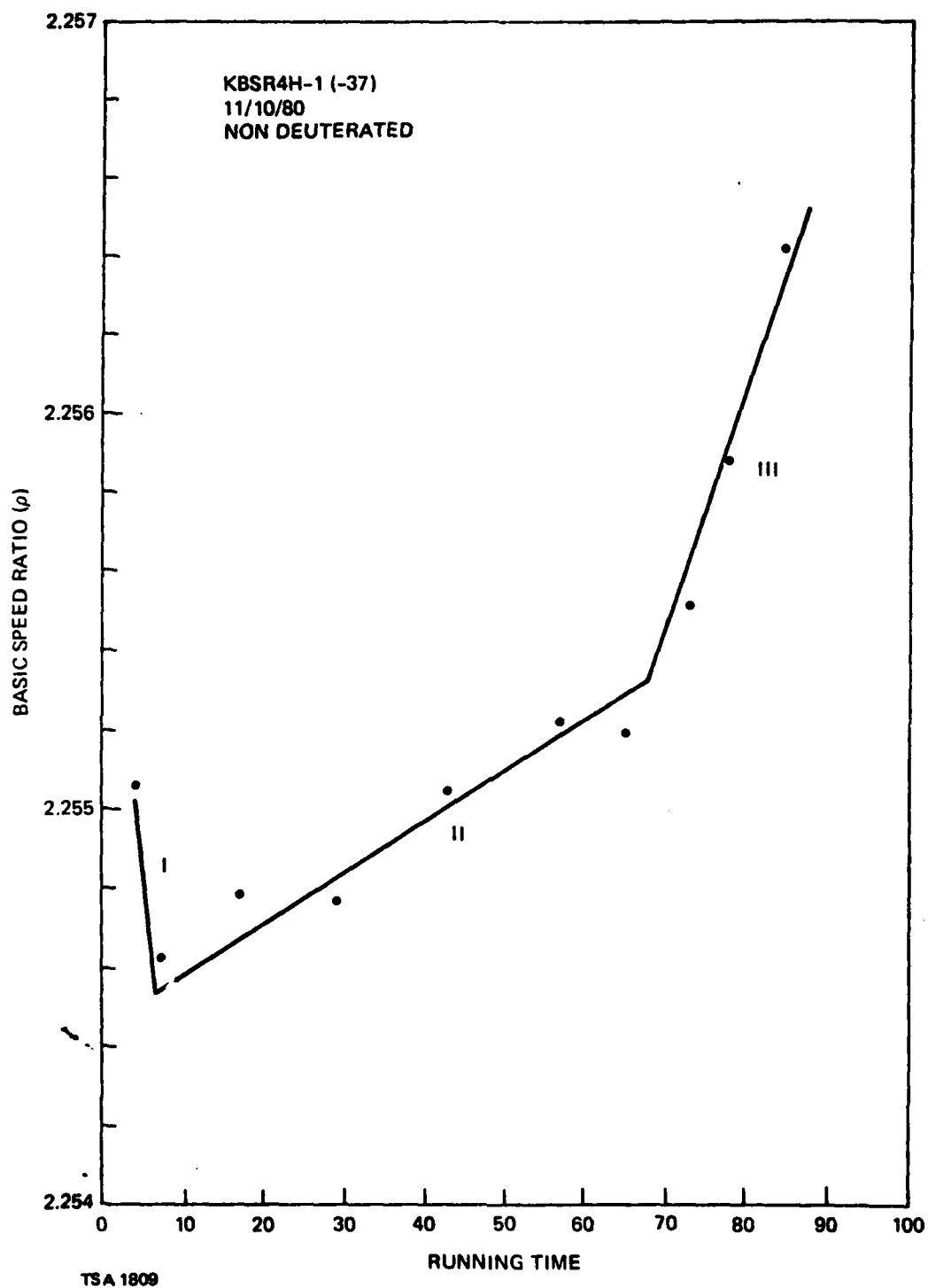


Figure 2-2. Starved testing basic speed ratio graph.

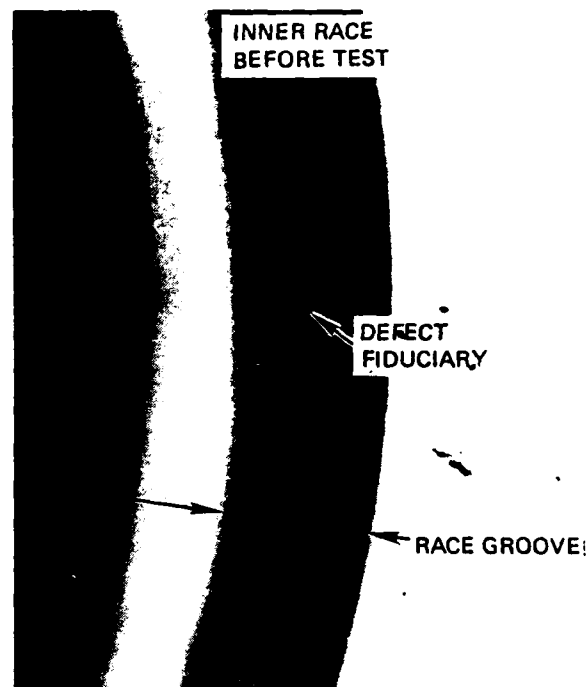
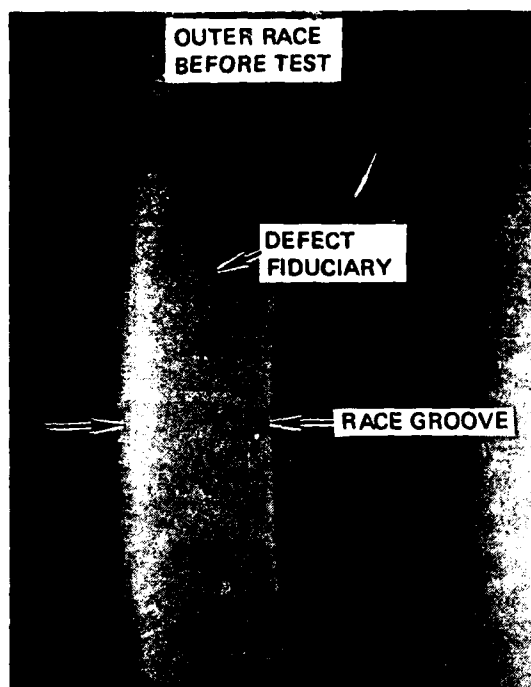
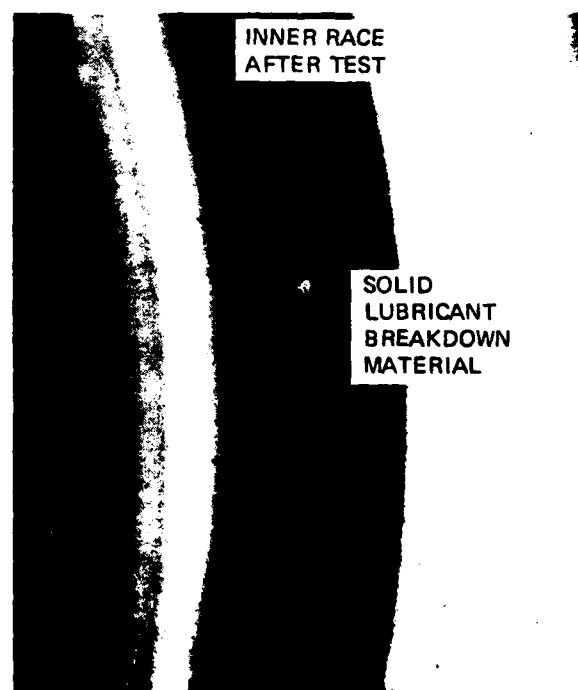
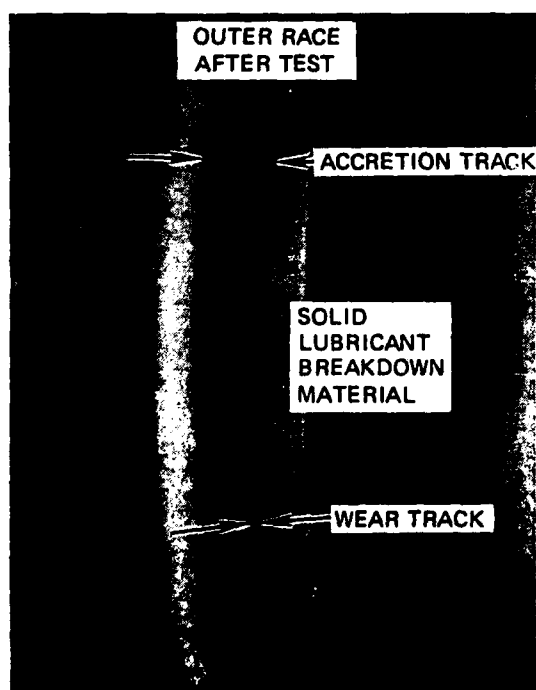


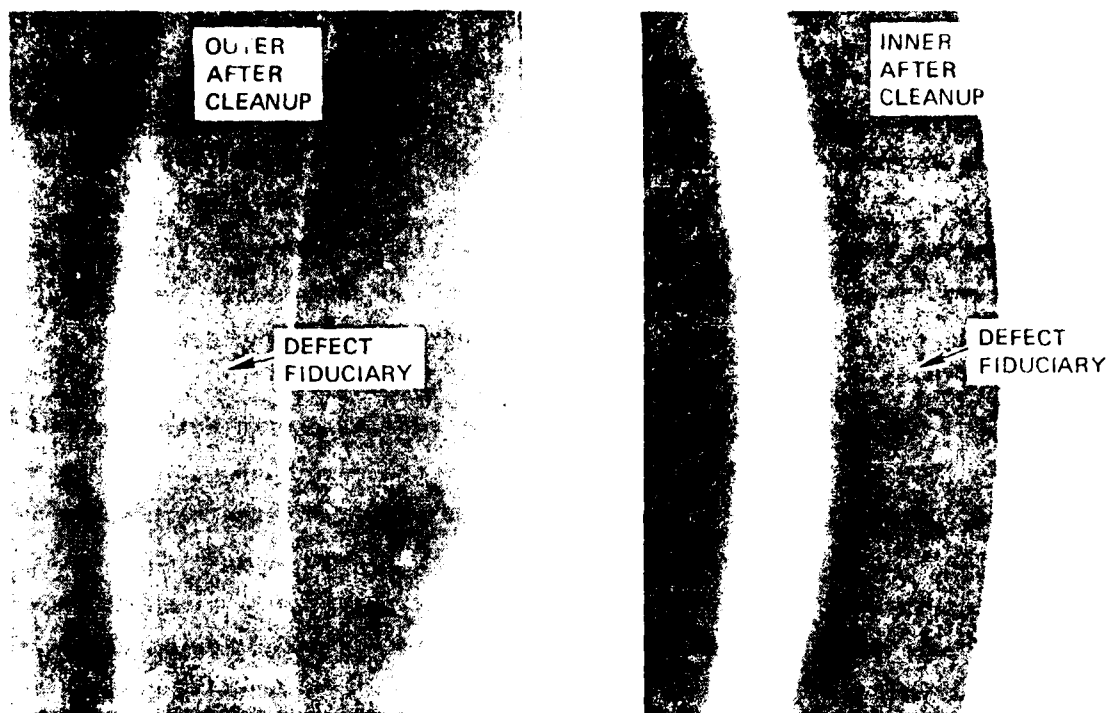
Figure 2-3(a). Races before lubricant failure.



TSA 1810

100×10^{-6} gm NON DEUTERATED

Figure 2-3(b). Races after lubricant failure.



TSA 1811

Figure 1-1(c). Races after cleanup.

It was hoped to be able to show a statistically significant difference in the time spent in Region II for the two oils as a measure of their resistance to degradation. Initial oil charge is the critical independent variable in these tests. It was possible to supply small (100×10^{-6} gm) amounts of oil more or less reproducible to the ball sets by withdrawing each ball from a diluted solution of oil in Freon and drying on a screen support before assembly. The races were left dry after final cleaning. The effective initial charge in each test was indicated by the basic speed ratio measured at the end of Region I.

Nineteen such tests were run with different charges for the two oils; it was not possible to show any statistically significant difference between times to failure (averaging about 1 hour) although there was no problem in inducing failure with either material (Figure 2-3b). One unanticipated difficulty involved precession of the balls in some of the low charge tests. The definition of basic speed ratio in its original form assumes pure spin. It was not possible in the present program to expand the definition of basic speed ratio to include precession, or to explore its underlying causes in any detail.

Several objections to the test rationale were raised: Does the Freon (or any solvent) have an effect on the deposited oil? Is it the same chemically and physically as the bulk oil? Is the time spent in Region II really a measure of lubricant degradation resistance or is it more a measure of fluid mechanical flow properties? In view of these uncertainties and in the absence of unambiguous experimental differences it was decided to abandon this test series.

2.3.2 Flooded Tests

Questions of initial oil charge, precession, and adulterated lubricant are eliminated if the tests are carried out flooded. The trade-off is that the tests become much longer, no failure occurs, and no definite quantitative measure of degradation is easily available. However, it was thought that comparison of the accretion tracks left on the balls after a standard test would at least give a qualitative comparison of the two oils. This was eventually confirmed.

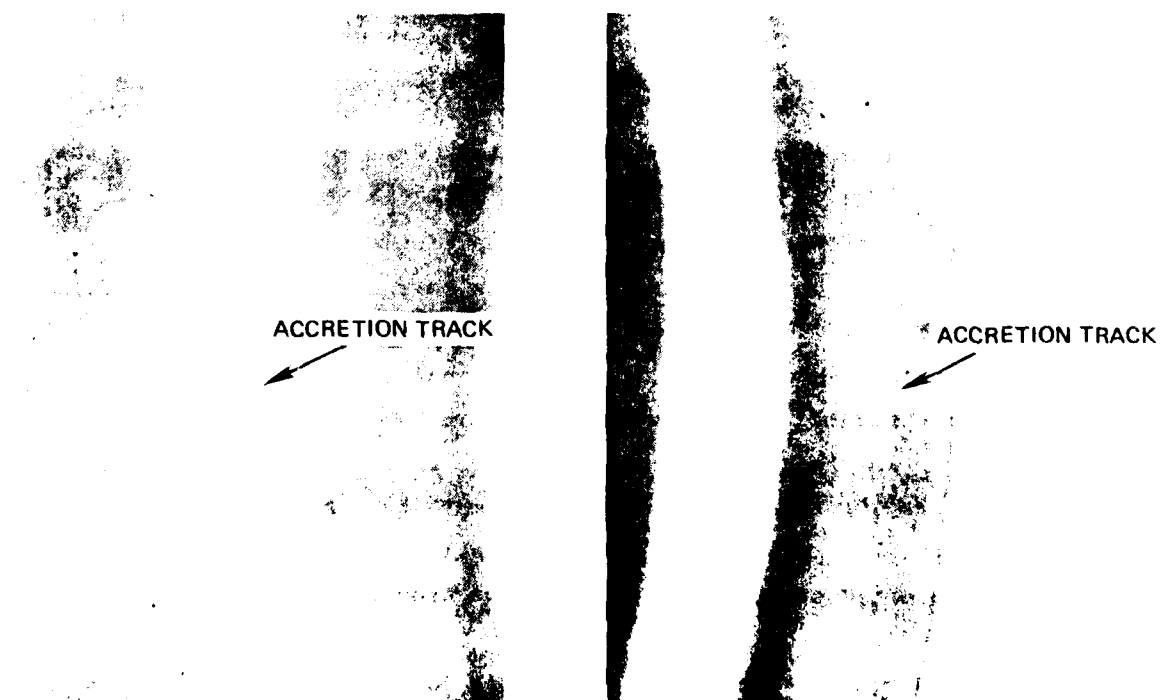
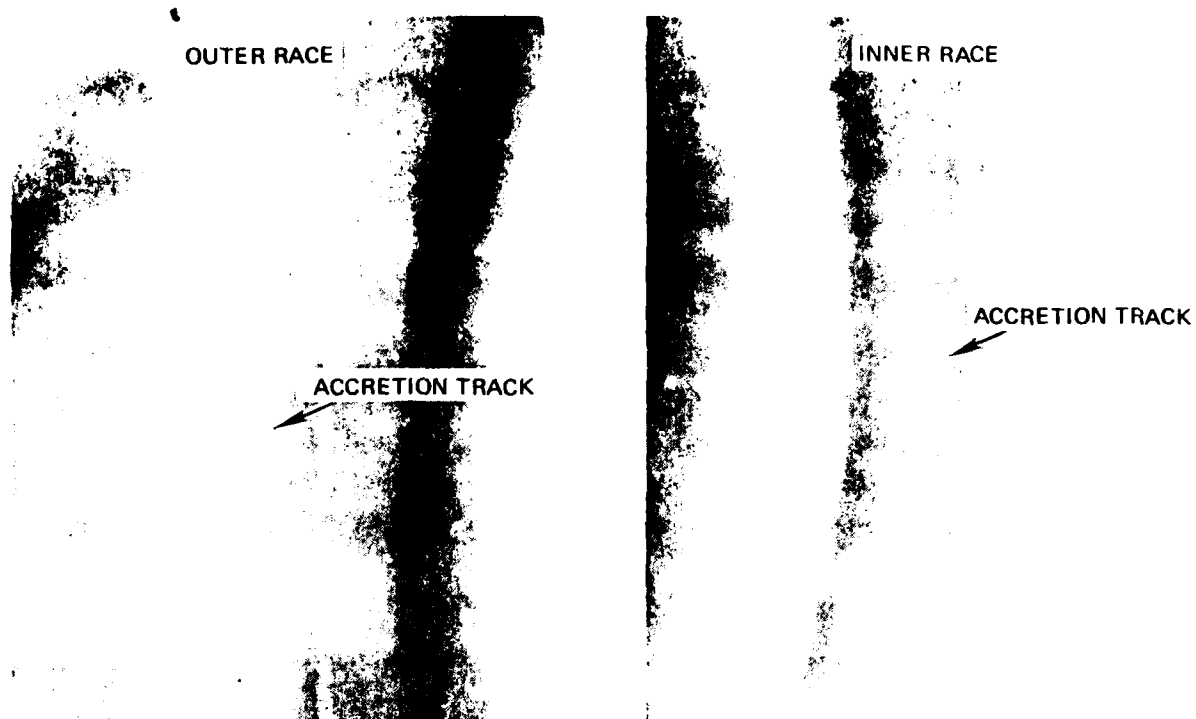
Accordingly, two "flooded" tests were run, each lasting 712 minutes, under the same conditions as for the starved tests (Table 2-1). The tests were interrupted at 300 minutes; the bearings were disassembled, solvent cleaned (no removal of breakdown material) and inspected; the bearings were reassembled and relubricated,

and the run was continued. Since a large excess of oil was present, the basic speed ratio was measured to be constant throughout these tests, including before and after the shutdown. Figure 2-4 shows the bearing parts after 712 minutes for the two oils.

Looking first at the races, very faint accretion tracks were produced on both races with both oils. No real difference in breakdown product formation is evident. However, compared with the product formed in the starved tests, these results confirm two parts of the shear activation hypothesis: (a) breakdown goes on at a small rate all the time in the most favorable conditions (Region II), and (b) breakdown is very much more rapid and lethal in Region III.

Figure 2-5 shows the tracks produced on the single bored ball from each ball set after 303 minutes and at the ends of the two tests. (The dark circle in the center of each picture is an artifact resulting from the diffuse light illumination system.) At 303 minutes a single track is seen with either oil. At 712 minutes one test shows a double track, the other a darker single track.

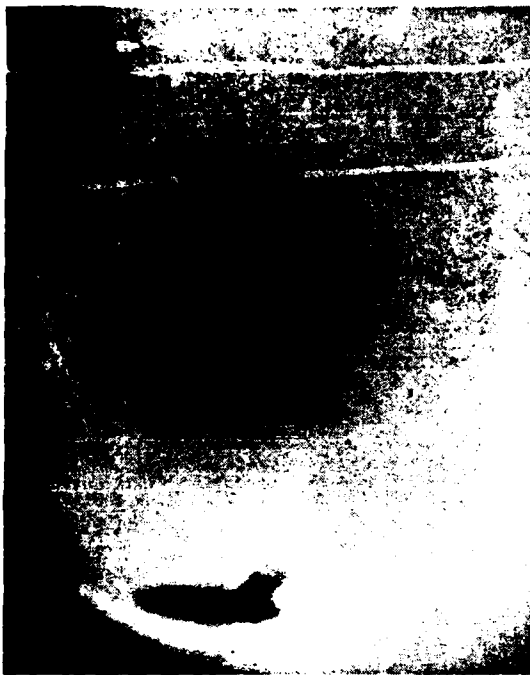
The double track was formed by chance when the test bearing was reassembled for the second half of the run with the bored ball inverted. It is difficult to say from these pictures whether the total product formed from the nondeuterated oil is more or less than that from the deuterated oil. However, when all the tracks formed on all the balls are compared (Figure 2-6) it is clear that substantially more came from the nondeuterated material. It is interesting to note that of the 28 balls shown in Figures 2-6, 12 show double tracks and were evidently inverted at the 303 minute reassembly. It must be noted, however, that these small circle tracks are the loci of ball-to-ball contacts, not ball-to-race contacts. Thus the degradation products in Figures 2-5 and 2-6 were formed in a low pressure-high slip EHD contact, as opposed to the products shown in Figure 2-4, which were formed in a high pressure-low slip EHD contact.



TSA 1812

DEUTERATED (712 minutes)

Figure 2-4. Lubricant degradation under flooded conditions on the races.



NON DEUTERATED (303 minutes)



DEUTERATED (303 minutes)



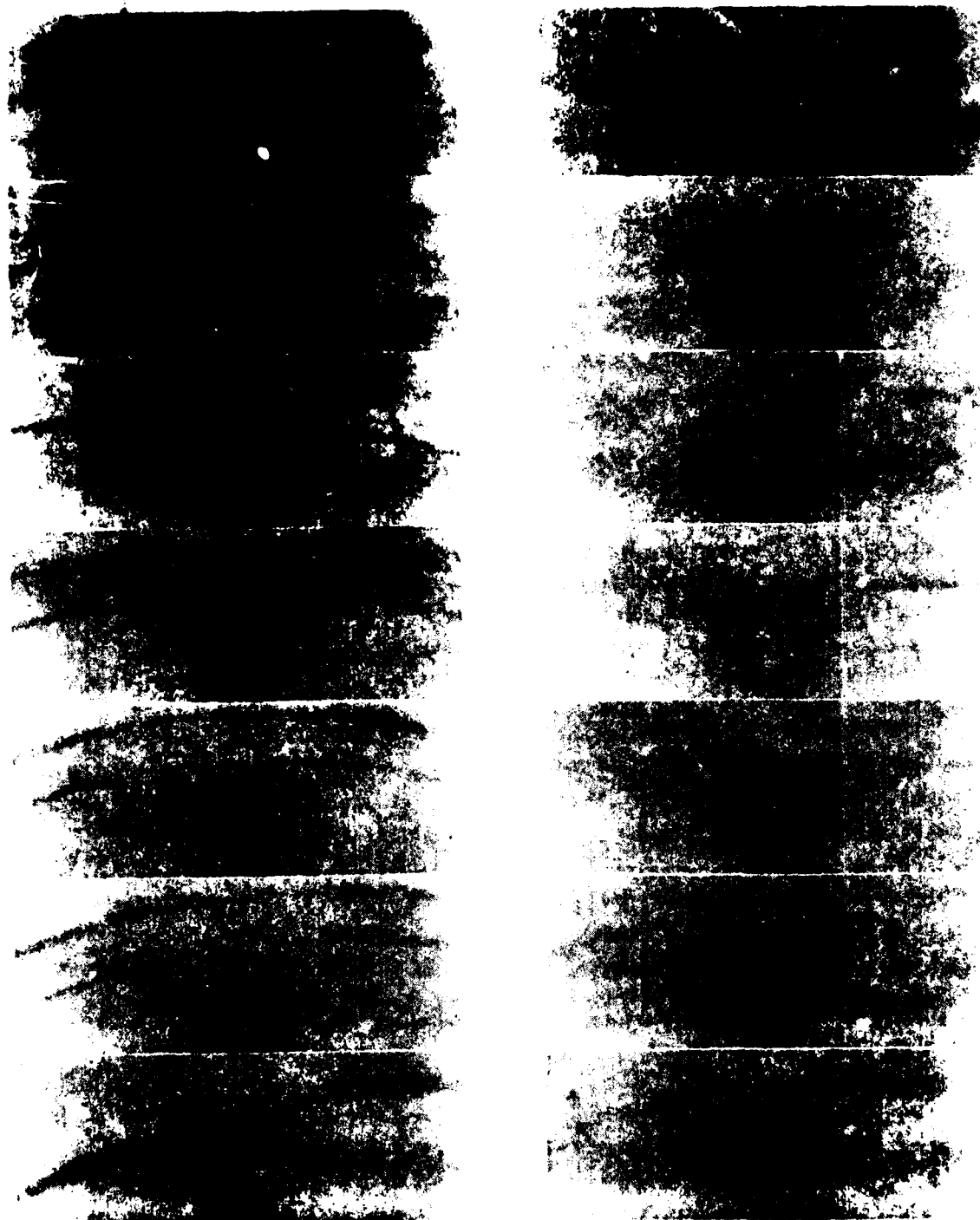
NON DEUTERATED (711 minutes)

TSA 1813



DEUTERATED (712 minutes)

Figure 2-5. Lubricant degradation under flooded conditions on the balls.

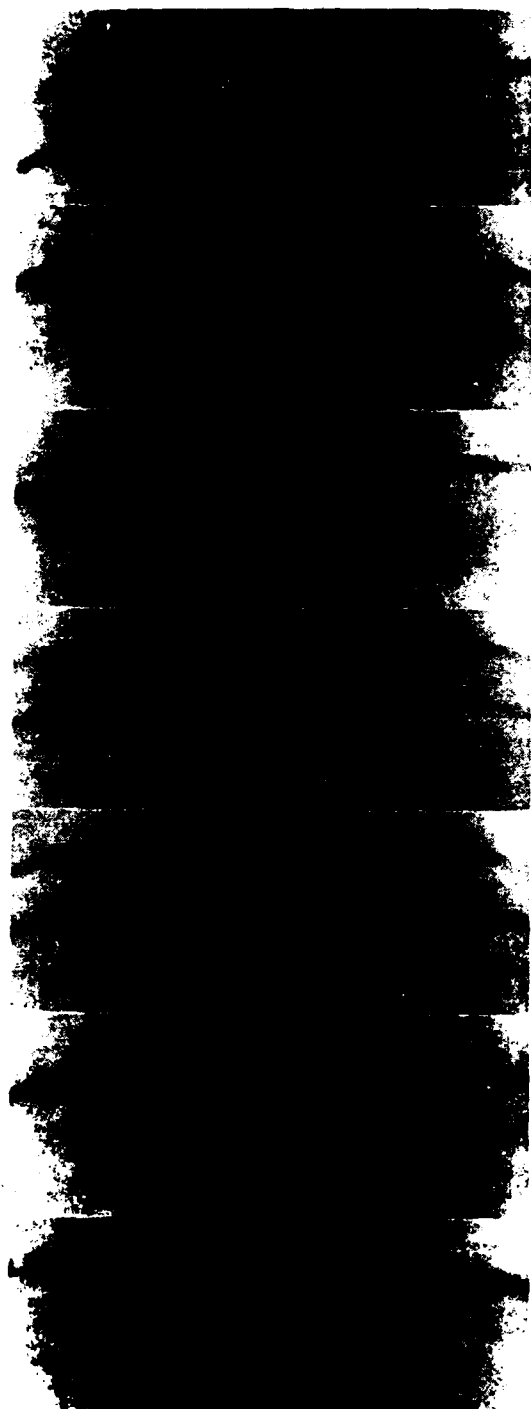


NON DEUTERATED

DEUTERATED

TSA 1814

Figure 2-3(a). Comparison of accretion tracks for all balls under flooded conditions.



NON DEUTERATED

TSA 1815



DEUTERATED

Figure 2-6(b). Comparison of accretion tracks for all balls under flooded conditions.

2.4 Conclusions

Nothing in these tests contradicts the shear activation hypothesis for lubricant breakdown reaction. However, the solvent deposition oil charge method is evidently not sufficiently sensitive to distinguish between the deuterated and hydrogenated lubricants. Besides the questions already discussed, solvent deposition does not give uniform oil coverage on the ball surface. Whether a particular ball spin axis gives a thin film on the rolling equator is thus determined statistically. These problems could be addressed by use of a delivery system to supply oil in known microgram increments in the wear track at known times.

As mentioned previously, when oil charge was reduced to obtain reasonable times to failure, ball precession often occurred with both the deuterated and hydrogenated oils (this has not been a problem with conventional mineral oils). With precession, basic speed ratio could not be measured, making it impossible to monitor approaching failure. Subsequently, in other programs, it has been shown that ball spin rate should be replaced by the quantity $(S + p \cos \theta)$ in the definition of basic speed ratio. S and p are the magnitudes of the (oblique) spin and precession components of ball angular velocity vector, separated by the angle θ , all of which can be measured. In principle, therefore, precession can be accounted for in these experiments. It is believed that the starved in situ test for lubricant breakdown susceptibility, while not completely successful in the present series, can be improved to give quick comparisons between lubricants. The question remains as to why these synthetic hydrogenated and deuterated test oils promote precession when conventional oils do not.

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